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EUROPEAN SCIENTIFIC NOTES

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SWEDISH REACTOR BECOMES CRITICAL

On 16 July 1954 the reactor of the Aktiebolaget Atomenergi, Stockholm, came into operation for the first time. This reactor is very similar in design to the old CP3 at the Argonne Laboratory and to the JEEP reactor in Norway. It contains 126 rods of aluminum-clad uranium each 1" in diameter and 2 meters long. The moderator consists of some 7 tons of heavy water which is circulated through the reactor and an air-cooled heat exchanger.

Initial operation of the reactor was kept at a power level of 0.2 watts which was the minimum power for reliable measurements and control. The method of approaching criticality was to put in more uranium than calculations indicated was necessary, then gradually to raise the level of the heavy water in the tank until the reaction became self maintaining. Extensive tests were conducted on the effect of the control and safety rods. It is planned to maintain the reactor at the low power level of 0.2 watts for a period of about six months. At the end of this time it is planned to dismantle the reactor and inspect the various components for possible damage including, of course, any possible changes in the aluminum cladding of the uranium fuel rods. Depending upon the results of this inspection the reactor power may then be increased to the design value of 100 - 300 kw.

The Aktiebolaget Atomenergi is an independent corporation owned four-sevenths by the Government of Sweden and three-sevenths by private industry. The director of the corporation is Dr. H. Brynielson, a chemical engineer.

The reactor has been placed some 120 ft underground in a large chamber blasted out of solid rock. The protection afforded by this situation has been considered suf-

ficient to allow the reactor to be placed about a mile from the main shopping district of Stockholm. However, this underground location has also presented an extra condition on the safety aspects of the design which has required rather more elaborate precautions than is usually necessary. The maximum reactor power which is at present considered to be consistent with safety is 100 kw but it is believed that this will probably be increased to 300 kw after operating experience has been gained.

THERMOCHEMISTRY OF REDISTRIBUTION REACTIONS

A detailed analysis of the thermochemistry of a large number of "redistribution reactions" enabled Dr. H. A. Skinner (Manchester) to draw some valuable conclusions concerning conjugation and hyperconjugation in the molecules involved and also to obtain some subtle stereochemical inferences. A particularly simple example can be written as

$$A - X - A + B - X - B \longrightarrow A - X - B + A - X - B$$
.

The extent to which such reactions (in the gas phase) are not thermoneutral indicates the significance of bond energy variations in different compounds. Available tables of heats of formation are used in deriving the heats of reaction of interest. A detailed study is to appear in Rec. Trav. Chim. shortly and only a few of the more noteworthy conclusions are discussed here.

The isomerization of 1,4-pentadiene (g) to 1,3-pentadiene (g), $\Delta H = -6.60$ Kcal/mole, is readily explicable in terms of w-conjugation between the double bonds in the 1,3-pentadiene; on the other hand, the isomerization of n-pentane (g) to neopentane (g), $\Delta H = -4.67$ Kcal/mole is more difficult to interpret.

The difference between the values of $\Delta\,{\rm H_{r}}$ in the following two reactions:

1/2 CH₂ = CH - CH = CH₂ + 1/2 C₂H₆
$$\longrightarrow$$
 CH₃CH = CH₂ (1) \triangle H_r = 1.83

$$1/2 \text{ C}_{6\text{H}_5} \cdot \text{C}_{6\text{H}_5} + 1/2 \text{ C}_{2\text{H}_6} \longrightarrow \text{C}_{6\text{H}_5} \text{C}_{\text{H}_3} \dots$$
 (2) $\Delta \text{H}_r = 0.77$

may be a direct indication of the fact that since diphenyl is not coplanar, there is much less π -conjugation across its central bond than there is in 1,3-butadiene. On the other

hand similar considerations suggest that dibenzyl is abnormally stabilized to the extent of 4 Kcal/mole, possibly due to π -hyperconjugation. It should be remembered, however, that these conclusions depend on highly precise and reliable thermal data.

Comparing

 $1/2 \text{ CH}_3\text{CCCCH}_3 + 1/2 \text{ C}_2\text{H}_6 \longrightarrow \text{CH}_3\text{CCCH}_3; \triangle \text{H}_r = -2.7 \text{ Kcal/mole}$

with reaction (1) above suggests that there is much less conjugation between the C = 0 - groups in diacetyl, than between the CH2 = CH - groups in butadiene. Available dipole moment data indicates that diacetyl is probably a mixture of s-cis and s-trans rotational isomers at room temperature and this may support the conclusion reached from the thermal data.

DEHYDROGENATION UNDER MILD CONDITIONS

The dehydrogenation of hydroaromatic systems by quinones has been known for many years. Probably the most familiar of the quinones used for this purpose is tetrachloro-phenzoquinone (chloranil), but the use of quinones as a practical dehydrogenation method has not enjoyed the popularity of other methods, for example, those employing catalytic dehydrogenation, sulfur or selenium. All of these techniques normally employ vigorous conditions, including temperatures in the range of 200°-300°. In many instances a dehydrogenation method operable under mild conditions would be of interest.

Prof. R. P. Linstead and Dr. E. A. Braude of Imperial College, London, have recently developed some unusually reactive quinones which are capable of effecting dehydrogenation nearly quantitatively in refluxing benzene solution. The following three have been found to be among the most active

dehydrogenating agents, and they are all capable of dehydrogenating dihydro and tetrahydro naphthalene derivatives at relatively low temperatures.

All three of these quinones have high xidation-reduction potentials, a feature which apparently contributes to their activity as dehydrogenating agents. Preliminary experiments indicate that the reaction is probably a polar one and is catalyzed by acid.

A NEW SYNTHESIS OF ETHYLENE DERIVATIVES

An unusual new reaction with considerable potential synthetic value has been developed very recently by Prof. G. Wittig of the Chemical Institute of the University of Tübingen. The reaction involves ylides, an unusual type of compound which was discovered in Prof. Wittig's laboratory. The reaction can be illustrated by the following specific examples but it appears to be of a very general type. The key compound is an ylide derived from phosphorus and is prepared by quaternizing triphenylphosphine with methyl iodide, followed by treatment with phenyllithium. The ylide reacts at room temperature (in one or two hours) with ketones to give the corresponding ethylene derivatives and triphenylphosphine oxide.

$$(1) \quad (C_{6}H_{5})_{3}P + CH_{3}I \longrightarrow \left[(C_{6}H_{5})_{3}PCH_{3} \right]^{+}I \xrightarrow{C_{6}H_{5}L_{1}}$$

$$\left[(C_{6}H_{5})_{3}P \xrightarrow{\bigoplus} - CH_{2} \longleftrightarrow (C_{6}H_{5})_{3}P = CH_{2} \right] + \left[(C_{6}H_{5})_{3}P - CH_{2} \right] + \left[(C_{6}H_{5})_{3}PC + (C_{6}H_{5})_{3}PC \right] + \left[(C_{6}H_{5})_{5}PC + (C_{6}H_{5})_{5}PC \right] + \left[(C_{6}H_{5})_{5}PC +$$

It is particularly notable that cyclohexanone gives methylene cyclohexane in $80^{\circ}/_{\circ}$ - $90^{\circ}/_{\circ}$ yield and no methyl cyclohexene, the thermodynamically more stable form. Prof. Wittig and his group are studying extensions of the reaction to include a variety of ketones and they also found it possible to prepare benzal derivatives by substitution of benzyl chloride for methyl iodide in the preparation of the original reagent.

Since the ylides react rapidly with oxygen and moisture the reagent is prepared and the synthesis is conducted in Schlenk tubes under an inert atmosphere.

CONFERENCE ON DEFECTS IN CRYSTALLINE SOLIDS

A Conference on Defects in Crystalline Solids organized by the University of Bristol in cooperation with the International Union of Pure and Applied Physics, the Institute of Physics and the Physical Society, was held at the H. H. Wills Physical Laboratory in Bristol on 13 - 17 July. This Conference had a distinctly international flavor in that it attracted more than 300 participants from 18 different countries including Australia, Japan, India, South Africa, Israel and Canada, as well as the Western European nations and the United States. Some 50 papers were presented, most of which could be broadly classified into two main groups.

The opening portion of the Conference was devoted to a discussion of impurity centers and imperfections in solids as revealed particularly by microwave resonance techniques. This section included a general survey of the application of resonance techniques to the study of solids together with a number of papers on the nature and interactions of the defect centers in irradiated crystals. The latter half of the program was concerned largely with the properties of networks of dislocations in solids. Emphasis in this case was placed upon the chemical aspects of dislocations such as their influence on diffusion and precipitation processes, rather than on their relationship to plastic deformation. The mechanical effects of dislocations in solids formed the subject of a separate but complementary conference held at the University of Birmingham on 19 - 20 July (ESN 8, 221 (1954)).

The complete proceedings of the Bristol Conference will be published by the Physical Society. It is expected that a paper-bound report will be available before the end of the year at a price of about \$4.00; orders for the proceedings should be addressed to the Physical Society, 1 Lowther Gardens, London, S.W.7.

Two of the more interesting contributions dealing with the properties of dislocation networks are reviewed below; further details may be found in a forthcoming Technical Report ONRL-80-54.

Precipitation on Dislocations in Aluminum-Copper Alloys

Electron micrographic evidence for precipitation on dislocations in dilute Al-Cu alloys was presented by H. Wilsdorf (Pretoria, South Africa). A series of polycrystalline alloys containing from one to approximately five atomic percent Cu was investigated using techniques previously applied to Al, Cu and other metals (cf., Acta Met. 1, 394 (1953)).

With very lightly deformed specimens a series of faint and sometimes randomly-oriented surface traces was observed which did not appear to be associated with slip lines. These traces have been identified with single dislocations. Each section of a dislocation line is thought to give rise to a precipitate of one orientation only and the precipitates in these dislocations are assumed to be very closely spaced. On the basis of his observations, Wilsdorf feels justified in proposing that these traces represent networks of dislocations emitted by a source during plastic deformation. The average distance between the traces is about $3/4\mu$, which appears to be a reasonable value since the stress corresponding to such a distribution of dislocations amounts to approximately 1 kg/mm². Additional work is planned with single crystals.

Dislocations and Reactions in Solids

Dr. J. W. Mitchell (Bristol) continuing his important work on the separation of photolytic silver in crystals of AgBr (Hedges and Mitchell, Phil. Mag. 44, 223 (1953)), has demonstrated that dislocations may play an important role in chemical reaction mechanisms. He and one of his students, Evans, have exposed and developed coarse-grained photographic emulsions and then examined the AgBr crystals at high magnification under the electron microscope. It was found that with some modifications depending upon the intensity of exposure and the method of development, metallic silver is deposited only in certain isolated but closely-related regions within the crystal. A prolonged low intensity exposure always yielded a deposit of Ag in the center of each grain, this being the most highly distorted portion of the crystal. With chemical development, only one central spot was observed, but with more gentle physical development of the image, this central spot could be resolved into three separate specks arranged in a triangular pattern; in addition, Ag was also shown to deposit along sub-boundaries which extend from the center of the crystal to the centers of certain edges. High intensity exposures tended to activate sensitive regions around the edges of the crystal, but again the Ag was always found in positions which are geometrically related to one another, e.g. at the centers of edges 1200 apart.

The AgBr crystals in these emulsions always exhibited tabular forms with hexagonal outlines, even though the natural growth habit of the crystal is octahedral. All of the evidence obtained indicated that these tabular crystals grew from a dendritic nucleus whose thickness was approxi-

mately the same as the final thickness of the crystal (i.e. about 0.1 μ). It is believed that at a comparatively early stage, the dendrite becomes twisted about [111] in such a manner as to introduce a system of three screw dislocations (120° apart), which are co-planar with the dendrite. This system of screw dislocations makes possible the sidewise growth of the crystal and also seems to account for the preferential sensitivity of the crystal to photographic development.

Extremely thin filaments or whiskers of metallic Ag were also observed growing into the gelatine from certain points along the edges of the crystals. These filaments were always co-planar with the crystal and their points of emergence on the crystal edges always made an angle of 1200 with one another. It is probable that these filaments contain a single screw dislocation parallel to their axes, and that the filaments grow, in effect, by the deposition of metallic silver around the original screw dislocations responsible for the growth of the parent AgBr crystal. Certain chemical reagents have also been found to dissolve the crystal locally about the axes of the dislocations, leaving relatively deep notches in these special positions.

BIOCHEMICAL AND BIOPHYSICAL RESEARCH AT THE INSTITUT PASTEUR

At the Institut Pasteur, Dr. Jacques Monod has been promoted to position of Chief of Service in Biological Chemistry, filling the post left vacant by the death of Dr. Machel Previously Dr. Monod had been Chief of Laborator, Dr. A. Lwoff in the Department of Microbial Physiology.

Dr. Monod intends to continue his work on adaptive enzyme formation in bacteria. The enzyme used for study is β -galactosidase which is normally absent from E. coli. When thiomethyl galactoside is added to the medium, formation of β -galactosidase is induced. Thiomethylgalactoside is not split by the enzyme but serves simply as an inducer.

The β -galactosidase activity of a batch of cells is estimated by following colorimetrically the splitting of onitrophenylgalactoside. The splitting produces o-nitrophenol which has the characteristic yellow color. Resonance considerations prevent the o-nitrophenyl group from being colored in the galactoside.

Interest at the moment centers in the fact that removal of the inducer followed by culturing of the cells leads to little or no change in the <u>absolute</u> amount of β -galactosidase activity in a culture. Dr. Monod and his collaborators, Drs. Melvin Cohen and David Hogness (both from the United States), feel that an enzyme, once formed, undergoes little or no turnover or degradation. They disagree with those who hold that the materials of an existing enyzme can be used by a cell to construct other enzymes.

In Dr. Lwoff's department Drs. E. Wollman and F. Jacob are pursuing studies of the genetics of viruses infecting E. coli. Using dually tagged viruses they are studying crossover frequencies in an attempt to deduce the number of sexual contacts which occur. Dr. Lwoff has been in the United States studying techniques for culturing mammalian tissues. He intends to begin studies on viral infections of mammalian cells in tissue culture.

In the Biophysics Department (Chief of Service, Dr. Grabar), Dr. Curtis A. Williams, Jr., has extended the technique introduced by Dr. J. Oudin for bringing antigens and antibodies together by diffusion of the antibody into a gel column containing the antigen. Starting with an antigenic preparation which is suspected of being a mixture of two or more components, Dr. Williams injects this into either a rabbit or a horse, depending on the amount of material available. The antibodies when obtained are allowed to diffuse from the side into a flat agar block on which some of the antigen has been separated into various components by electrophoresis. Separate, crescent-shaped areas of precipitation appear if the components were electrophoretically separable. Even some crystalline proteins exhibit inhomogeneity by this technique.

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PERSONAL NEWS ITEMS

Dr. T. Suzuki of Tohuku University, Japan, is currently at the University of Bristol where he will spend the forthcoming year working in collaboration with Prof. F. C. Frank at the H. H. Wills Physical Laboratory.

Dr. J. D. McGee has resigned from his position as research physicist in the E.M.I. Research Laboratories to accept an appointment to the Chair of Instrument Technology in the University of London tenable at the Imperial College of Science and Technology.

Sir Robert Robinson, Professor of Chemistry at Oxford University, has been elected President of the British Association for the Advancement of Science. He succeeds Dr. E. D. Adrian, Master of Trinity College, Cambridge University.

TECHNICAL REPORTS OF ONRL

The following reports have been forwarded to ONR, Washington. Copies may be obtained by addressing requests to the Commanding Officer, Office of Naval Research Branch Office, Navy No. 100, c/o Fleet Post Office, New York, N.Y.

ONRL-59-54	"The Psychological Laboratory, The Universit of Stockholm" by W. D. Neff	У
ONRL-61-54	"Notes on Four European Tissue Culture Labor	a-

tories" by J. L. Tullis

ONRL-68-54 "Teaching and Research in Biology and Medicine in Dublin" by J. O. Hutchens

ONRL-69-54 "Malaria Reference Laboratory, Horton Hospital" by T. K. Ruebush

"Conference on Mechanical Effects of Dislo-ONRL-70-54 cations in Crystals" by L. Himmel

"Marine Biological Laboratories in France and ONRL-71-54 Monaco" by T. K. Ruebush

"Current Research in Guy's Hospital Dental ONRL-73-54 School, University of London" by G. L. Parke

"Insect Hormones and Eye Pigments" by J. C. ONRL-74-54 Sheehan

"Sterically Directed Syntheses" by J. C. Sheehan ONRL-75-54

ONRL-76-54 "Some Applications of Strong Focusing Fields at the Institute of Physics, University of Bonn" by J. R. Richardson

"Dehydrogenation Under Mild Conditions" by ONRL-77-54 J. C. Shehan

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